PATENT
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For: HIGH-DENSITY DETERGENT COMPOSITION

DECLARATION UNDER 37 CFR 1.132

Commissioner for Patents
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Sir:

I, Teruo KUBOTA, residing in Wakayama city, Wakayama prefecture, Japan, hereby declare and state as follows:

I am a graduate of Nagoya University, School of Engineering, Department of Chemical and Biological Engineering in the year 1993, majoring in chemical engineering. I have been employed in Kao Corporation since the year 1993 and have been assigned to the Research Laboratories. I have been involved in the research and development of powder process engineering since 1993.

I am familiar with the contents of U.S. Application Serial No. 09/889,497, filed on July 18, 2001, entitled HIGH-DENSITY DETERGENT COMPOSITION, its prosecution before the United States Patent and Trademark Office, and the references cited therein.

The following experiments were carried out by me directly and/or under my direct supervision:

Base detergent granules were prepared in accordance with Preparative Examples 1, 2, 3, and 4 of the above-identified application. Some base detergent granules of each of Preparative Examples 1-4 were used as comparative examples in this report. Some base detergent granules from each of Preparative Examples 1-4 were subjected to particle size distribution adjustment in accordance with procedures described in the above-identified application. For the sake of consistency with Table 2 in the specification, the classified samples of Preparative Examples 1-4 are referred to herein as Examples 1, 5, 8, and 10, respectively.

The "before classification" and the "after classification" particles were subjected to Evaluation 1 (dissolubility of detergent), Evaluation 2 (dispersibility of detergent), Evaluation 3 (detergency of detergent), and Evaluation 4 (hand-

washing dissolution), as those evaluation tests are described on pages 26-29 of the application.

The experimental results are summarized in the following Tables:

Base detergent granules from Preparative Example 1	*Before classification*	*After classification (Example 1)*
W [more than 2000μm]	0.06	
W [1410-2000μm]	0.05	0.00
W [1000-1410μm]	0.08	0.00
W [710-1000μm]	0.12	0.00
W [500-710μm]	0.14	0.01
W [355-500μm]	0.17	0.13
W [250-355μm]	0.15	0.40
W [180-250μm]	0.12	0.40
W [125-180μm]	0.08	0.04
W [less than 125μm]	0.04	0.02
Average particle size [μm]	450	259
Bulk density [g/L]	761	773
Flowability [sec]	6.7	6.3
$S(W_i-V_i)$ [%]	81.4	99
Evaluation 1	D	A
Evaluation 2	I	I
Evaluation 3	42	54
Evaluation 4 [sec]	210	36

Base detergent granules from Preparative Example 2	*Before classification*	*After classification (Example 5)*
W [more than 2000μm]	0.01	
W [1410-2000μm]	0.03	0.01
W [1000-1410μm]	0.11	0.02
W [710-1000μm]	0.22	0.06
W [500-710μm]	0.26	0.07
W [355-500μm]	0.15	0.16

W [250-355μm]	0.10	0.40
W [180-250μm]	0.05	0.18
W [125-180μm]	0.04	0.08
W [less than 125μm]	0.03	0.02
Average particle size [μm]	596	303
Bulk density [g/L]	808	821
Flowability [sec]	6.6	6.8
$S(W_i-V_i)$ [%]	81.3	95
Evaluation 1	D	B
Evaluation 2	I	II
Evaluation 3	43	48
Evaluation 4 [sec]	295	95

Base detergent granules from Preparative Example 3	*Before classification*	*After classification (Example 8)*
W [more than 2000μm]	0.00	
W [1410-2000μm]	0.02	0.00
W [1000-1410μm]	0.09	0.00
W [710-1000μm]	0.21	0.04
W [500-710μm]	0.20	0.09
W [355-500μm]	0.14	0.25
W [250-355μm]	0.11	0.23
W [180-250μm]	0.08	0.19
W [125-180μm]	0.06	0.10
W [less than 125μm]	0.09	0.10
Average particle size [μm]	521	296
Bulk density [g/L]	766	772
Flowability [sec]	6.7	6.7
$S(W_i-V_i)$ [%]	81.6	95.6
Evaluation 1	D	B
Evaluation 2	I	II
Evaluation 3	44	52
Evaluation 4 [sec]	326	90

Base detergent granules from Preparative Example 4	*Before classification*	*After classification (Example 10)*
W [more than 2000μm]	0.04	
W [1410-2000μm]	0.03	0.00
W [1000-1410μm]	0.06	0.00
W [710-1000μm]	0.15	0.03
W [500-710μm]	0.17	0.10
W [355-500μm]	0.18	0.26
W [250-355μm]	0.15	0.30
W [180-250μm]	0.12	0.21
W [125-180μm]	0.06	0.08
W [less than 125μm]	0.04	0.02
Average particle size [μm]	454	312
Bulk density [g/L]	860	873
Flowability [sec]	6.2	6.0
$S(W_i-V_i)$ [%]	91.1	99.2
Evaluation 1	C	A
Evaluation 2	I	II
Evaluation 3	47	52
Evaluation 4 [sec]	122	29

In Evaluation 1, which measures dissolubility of a detergent in a washing machine, particle size adjustment in accordance with the present invention has improved dissolubility by at least two grades (the grades are explained on page 26 of the specification), from D to A in Example 1, from D to B in Example 5, from D to B in Example 8, and from C to A in Example 10. In Evaluation 4, hand-washing dissolubility, particle size adjustment in accordance with the present invention has likewise been shown to improve dissolubility. The adjusted particle size profile granules of Example 1 take only 17% as much time to dissolve as do the unadjusted particles of Preparative Example 1. The adjusted particle size profile granules of Example 5 take

only 32% as much time to dissolve as do the unadjusted particles of Preparative Example 2. The adjusted particle size profile granules of Example 8 take only 28% as much time to dissolve as do the unadjusted particles of Preparative Example 3. The adjusted particle size profile granules of Example 10 take only 24% as much time to dissolve as do the unadjusted particles of Preparative Example 4.

For the sake of comparison, the following data relating to the Van Dijk WO 94/02573 disclosure is also presented:

A base powder is prepared in the form of agglomerates using a Loedige batch mixer. The agglomerates were made by placing into the mixer/granulator a mixture of powders consisting of: 23.8 kg of zeolite A (average particle size 3.5 μm); 4.8 kg sodium carbonate; 0.4 kg optical brightener; and 1.0 kg magnesium sulfate. With the mixer/granulator operating, 3.6 kg of an aqueous solution of tripolyphosphonate was added to the powder mixture. Immediately thereafter, 19.1 kg of a high activity anionic surfactant paste at 50°C was poured into the mixer/granulator. The high activity surfactant paste had a total anionic surfactant activity of 76% and a water content of 20%. The surfactant in the paste consisted of a mixture of C_{12}- and C_{14}-alkyl sulphates (AS) and C_{12}- and C_{14}-alkyl ether sulphates with an average of 3 moles of ethylene oxide (AE3S), in the weight ratio of AS:AE3S of 80:20. The agglomerates so

made were then dried in a fluid bed with an inlet air temperature of 90°C. The resulting agglomerates are referred to as the base powder.

40 kg of this base powder was sieved through a Tyler sieve mesh 10 to remove the coarse fraction (> 1700 microns), and on a Tyler sieve mesh 65 in order to remove the fines (< 212 microns). The remaining fraction was then used to prepare a detergent composition containing:

Base powder (after sieving)	32 kg
Acrylic-maleic copolymer (granular)	2.4 kg
Layered silicate (granular)	15.1 kg
Perborate monohydrate	17 kg
Bleach activator (agglomerated)	7.3 kg
Suds suppressor (agglomerated)	2.0 kg
Emulgen103 (trademark, Kao)	5.7 kg
Perfume	0.2 kg
Dobanol (surfactant)	2.5 kg
Zeolite A	3.6 kg

This composition was made by mixing all of the dry components (except the zeolite) and evenly distributing the Dobanol surfactant and perfume onto the mixture by spraying. Then the zeolite A powder was added and allowed to mix for 3 minutes. Finally the batch was split into two equal portions and mixed with the filler particles (in this example, the filler particles were sodium carbonate). Half of the batch (44 kg) was mixed with 6 kg ground carbonate with an average particle size of 175 microns. The ground carbonate was classified and remixed having

the following particle size distributions (expressed cumulatively in weight-%) before being mixed with the detergent composition:

	Ground carbonate
On Tyler 14 (> 1180 microns)	0
On Tyler 20 (> 850 microns)	0
On Tyler 35 (> 425 microns)	0
On Tyler 65 (> 212 microns)	19
On Tyler 100 (> 150 microns)	62
Weight fraction < 150 microns	38%

The following Table shows how such particles prepared in accordance with the teachings of Van Dijk WO 94/02573 perform in various tests applied by Applicants to their own particles hereinabove:

Base detergent granules (van Dijk comparison)	
W [more than 2000µm]	0.00
W [1410-2000µm]	0.66
W [1000-1410µm]	8.45
W [710-1000µm]	23.07
W [500-710µm]	19.00
W [355-500µm]	20.35
W [250-355µm]	17.12
W [180-250µm]	6.89
W [125-180µm]	1.90
W [less than 125µm]	2.56
Average particle size [µm]	511
Bulk Density [g/L]	781
Flowability [sec]	7.1
$S(W_i-V_i)$ [%]	63.8
Evaluation 1	D
Evaluation 2	IV
Evaluation 3	355
Evaluation 4 [sec]	34

Conclusion

As can be seen from the above-reported results, classification in accordance with the present invention (as it is reflected in the claims of the above-identified application) dramatically improves commercially significant properties of the detergent compositions in question. In my opinion, persons of ordinary skill in the art would not have expected improvements of the degree illustrated by the above experimentation due simply to particle size adjustment in accordance with the invention of application Serial No. 09/889,497.

The undersigned declares further that all statement made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statement may jeopardize the validity of above identified application or any patent issuing thereon.

July 26, 2005
Date

Teruo Kubota
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